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AEC RESEARCH AND DEVELOPMENT REPORT.

# INSTRUMENTAL METHODS OF URANIUM ANALYSIS

Y-12 LABORATORY DEPARTMENT

**UNION CARBIDE NUCLEAR COMPANY**
DIVISION OF UNION CARBIDE CORPORATION

*Operating*

- OAK RIDGE GASEOUS DIFFUSION PLANT
- OAK RIDGE Y-12 PLANT
- OAK RIDGE NATIONAL LABORATORY
- PADUCAH GASEOUS DIFFUSION PLANT

*for the Atomic Energy Commission*

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# INSTRUMENTAL METHODS OF URANIUM ANALYSIS

Y-12 Laboratory Department

Oak Ridge, Tennessee
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# INSTRUMENTAL METHODS OF URANIUM ANALYSIS

Y-12 LABORATORY DEPARTMENT

## ABSTRACT

Five analytical procedures are reported which give, in detail, the instrumental methods used at Y-12 for uranium analysis. They are: (I) Determination of $U^{+4}$ in Uranium Oxides and Uranium Tetrafluoride by Potentiometric Titration with Potassium Dichromate, (II) Colorimetric Determination of Uranium, (III) Isotopic Analysis of $U_3O_8$ Using the Optical Spectrograph, (IV) High Precision Determination of Uranium by Potentiometric Titration with Potassium Dichromate, and (V) Fluorimetric Determination of Low Concentrations of Uranium by Extraction and Dilution Methods.

# CONTENTS

## PROCEDURE I - DETERMINATION OF $U^{+4}$ IN URANIUM OXIDES AND URANIUM TETRAFLUORIDE BY POTENTIOMETRIC TITRATION WITH POTASSIUM DICHROMATE

### INTRODUCTION

This method is used for determining uranium that is in the +4 valence state in uranium oxides and uranium tetrafluoride or a mixture of the two. It is based upon the quantitative manner in which uranium oxides and tetrafluoride will dissolve in ortho-phosphoric acid without a change in the valence state of the uranium. Since reduced uranium solutions oxidize easily when in contact with the atmosphere, the sample is kept under carbon dioxide gas from the time the solution is started until the titration is completed.

Using this method, samples containing 0.6 gram to 0.8 gram of $U^{+4}$ can be analyzed with a precision of + 0.2 percent. Samples determined without using carbon dioxide gas yielded results which averaged 1.0 percent low.

The determination consists of: (1) dissolving the sample in concentrated phosphoric acid, (2) diluting with 7.5% sulfuric acid, (3) adding excess dichromate solution, and (4) titrating the excess dichromate with ferrous ammonium sulfate solution.

The sample is titrated in a cell containing a platinum electrode and a calomel reference electrode. Since a uranium-dichromate couple does not produce a sharp change of potential, an iron-dichromate potential change is used. The iron-dichromate couple produces a change in potential of 200 to 300 millivolts.

It is apparent from the foregoing that any reduced elements that are oxidized by dichromate will yield high results. Reduced iron is the major interference in the production samples. Correction for iron in $UO_2$ and $UF_4$ samples is made either from spectrographic iron results or from a colorimetric determination of iron since it is assumed that the iron is in a reduced state in these samples. No correction for iron is necessary in $UO_3$ and $U_3O_8$ samples.

### REAGENTS

Carbon Dioxide Gas, cylinder
Ferrous Ammonium Sulfate Solution, ~0.05 N in 5% $H_2SO_4$
Potassium Dichromate Solution, 0.1500 N
Phosphoric Acid, concentrated
Sulfuric Acid, 7.5% by volume

## EQUIPMENT

Burettes, Normax, 10 mls and 50 mls
Flask, Erlenmeyer, (wide mouth), 500 mls
Titration Assembly, see Figure 2, Page 12.

## PREPARATION AND STANDARDIZATION OF SOLUTIONS

### Potassium Dichromate Solution

Compounds - $K_2Cr_2O_7$ (primary standard) - 7.3556 grams; distilled water - ~1 liter.

The potassium dichromate solution is prepared by weighing 7.3556 g of primary standard potassium dichromate to the nearest 0.1 mg and diluting with distilled water to one liter in a volumetric flask.

### Ferrous Ammonium Sulfate Solution

Compounds - $Fe(SO_4)\cdot(NH_4)_2SO_4\cdot 6H_2O$ (cp crystals) - 157 g; $H_2SO_4$ (concentrated) - 80 ml; distilled water - 2 liters.

The ferrous ammonium sulfate solution is prepared by adding 80 mls of concentrated sulfuric acid to 2000 mls of distilled water. When the mixture is cool, 157 grams of ferrous ammonium sulfate is added and then diluted with distilled water to about 8 liters in a carboy. The solution is stirred until thoroughly mixed.

### Standardization of Ferrous Ammonium Sulfate Solution

The ferrous ammonium sulfate solution is standardized against the standard potassium dichromate solution. Twelve to fifteen mls of potassium dichromate solution is added to approximately 200 mls of 7.5% sulfuric acid and titrated with ferrous ammonium sulfate solution until the end point is reached.

The ferrous ammonium sulfate solution is standardized daily since the solution is not protected by $CO_2$.

Formula for ferrous factor:

$$\text{Ferrous factor} = \frac{\text{mls of potassium dichromate solution}}{\text{mls of ferrous ammonium sulfate solution}}$$

# PROCEDURE

## Sample Preparation

The entire sample is ground until it will pass through a 60-mesh sieve and rolled until thoroughly mixed.

Duplicate aliquants of the sample, each of which will contain 0.6 to 0.8 g of $U^{+4}$, are weighed and the net weights are recorded on the work card. Subsequently, each aliquant is transferred to a 500-ml wide mouth Erlenmeyer flask appropriately identified with the requisition number and the aliquant.

Thirty to thirty-five mls of concentrated phosphoric acid is added to the flask. The acid is not added, however, until the flask is ready to be placed on the hot plate since some of the sample will dissolve while in contact with the atmosphere.

The flask is placed on the hot plate with carbon dioxide gas over the sample until the sample is completely dissolved, see Figure 1, Page 12.

When all the sample is completely dissolved, the flask is removed from the hot plate and the sample is cooled. Then the sample is diluted with approximately 200 mls of 7.5% sulfuric acid. The $CO_2$ gas must cover the sample from the time the solution is started until the titration is completed.

## Titration

After the sample is placed in the titration assembly (Figure 2, Page 12), the millivoltmeter is turned on. The meter will read in the range of +200 to +300 millivolts.

Using the 50-ml Normax burette, standard potassium dichromate solution is added until the meter reads +700 to +750 millivolts. This insures complete oxidation of the uranium plus an excess of dichromate. The burette reading is recorded on the work card.

With the 10-ml Normax burette, ferrous ammonium sulfate solution is added until a sharp drop in potential occurs. The potential should change 200 to 300 millivolts per drop of ferrous solution. The burette reading is recorded on the work card.

When the titration is completed, the sample solution in the flask is salvaged in the appropriate recycle U salvage container.

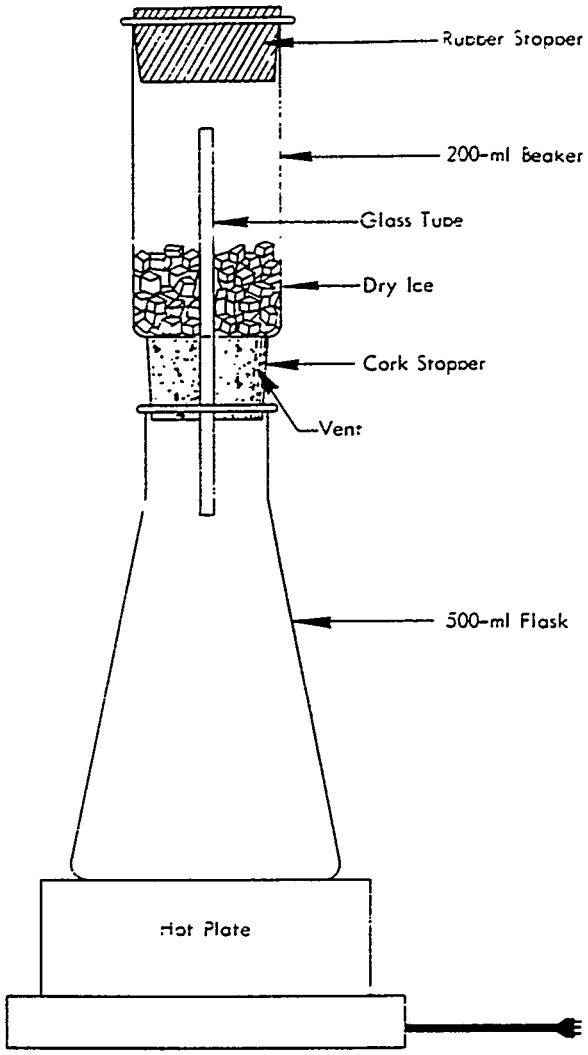



Figure 1. SAMPLE PREPARATION ARRANGEMENT.

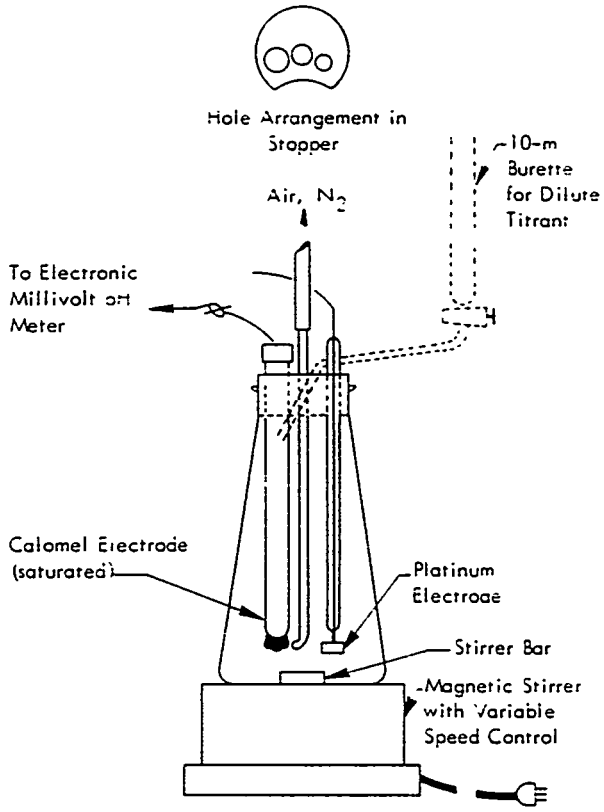



Figure 2. TITRATION ARRANGEMENT. (Potentiometric Titration)

## CALCULATIONS

### Grams of Uranium$^{+4}$ per Gram of Material

Formula 1 -

$$\text{ml excess } K_2Cr_2O_7 = (\text{ml } Fe(SO_4) \cdot (NH_4)_2SO_4 \cdot 6H_2O) \text{ (ferrous factor)}$$

Formula 2 -

$$\text{meq wt} = \frac{\text{at wt of element or mol wt of compound}}{(\text{valence change})(1000)}$$

Formula 3 -

$$\text{g } U^{+4}/\text{g} = \frac{(\text{total ml } K_2Cr_2O_7 - \text{ml excess } K_2Cr_2O_7)\ (N)\ (\text{meq wt})}{\text{grams of sample}}$$

Iron Corrections

For precise analysis of the gram uranium$^{+4}$ gram material, an iron correction is made. This correction factor is calculated from the spectrographic iron results or from a colorimetric determination of iron.

The iron correction from a colorimetric determination is determined by the following formulas:

Formula 1 -

$$\text{Fe correction} = (\text{g Fe/g})\ (\text{Fe equiv of U})$$

Formula 2 -

$$\text{corrected g } U^{+4}/\text{g} = \text{g } U^{+4}/\text{g} - \text{Fe correction}$$

Since the spectrographic iron result is expressed in parts per million of iron in uranium oxide, the iron equivalent of uranium must be determined in terms of uranium metal.

Briefly the formulas for iron correction from spectrographic iron results are:

Formula 1 -

$$1 \text{ ppm} = 0.000001 \text{ g/g}$$

Formula 2 -

$$\text{Fe (corrected)} = \frac{(\text{g } U^{+4}/\text{g})\ (\text{g Fe/g})}{0.4}$$

Formula 3 -

$$\text{corrected g } U^{+4}/g = \text{uncorrected g } U^{+4}/g - \text{Fe correction}$$

The complete calculations are checked to ascertain the accuracy.

SAMPLE WORK CARD

A typical sample analysis of a $U^{+4}$ determination is shown in Figure 3.

MISCELLANEOUS LABORATORY WORK CARD

| 500 | 11-9-57 | N-30 | 101-12-0713 | g $U^{+4}$/g | 111111 |
|---|---|---|---|---|---|
| RECYCLE | DATE | CONT NO | BATCH NUMBER | DETERMINATION | REQUISITION NUMBER |

| | | | | |
|---|---|---|---|---|
| Aliq. No. | | 1 | 2 | Remarks |
| Aliq. Wt. | By: ABC | 1.0220 g | 1.0482 g | N of $K_2Cr_2O_7$: 0.1500 |
| | TITRATION | | | $Fe^{+2}$ Factor: 0.3358 |
| $K_2Cr_2O_7$ | | 49.95 ml | 50.15 ml | |
| $Fe(SO_4) \cdot (NH_4)_2SO_4 \cdot 6H_2O$ | | 6.36 ml | 3.75 ml | Meq Wt. of U: 0.119039 |

CALCULATIONS

Aliq. No. 1

$$\frac{49.95 - (6.36 \times 0.3358) \times 0.1500 \times 0.119039}{1.0220} = 0.835311 \text{ g } U^{+4}/g$$

Report

Avg. g $U^{+4}$/g = 0.834070

Aliq. No. 2

$$\frac{50.15 - (3.75 \times 0.3358) \times 0.1500 \times 0.119039}{1.0482} = 0.832830 \text{ g } U^{+4}/g$$

Checked by: WF

LAB-99 (7-58)

Figure 3. WORK CARD SHOWING TYPICAL ANALYSIS DATA FOR URANIUM (IV).

## PROCEDURE II - COLORIMETRIC DETERMINATION OF URANIUM

### INTRODUCTION

Although there are a number of substances which will form colored compounds or complexes with uranium, the procedure used routinely in this laboratory takes advantage of the yellow-brown complex formed by uranium with l-ascorbic acid. The color is developed by hexavalent uranium in the presence of an excess of ascorbic acid in a buffered acid solution.

Under controlled conditions, the color intensity is a linear function of the uranium concentration in the range of 10-200 μg U/ml. The color intensity is a function of pH also in the ranges of 1 to 4 and 5 to 7, but it is independent of the pH in the range of 4.3 to 4.8; consequently, the routine procedure calls for a pH of 4.5 - 4.6. A pyridine-ammonium acetate-acetic acid solution is used for a buffer solution.

The pyridine in the buffer solution is primarily a complexing agent for copper, which interferes seriously with the determination. Very small amounts of chromium and vanadium, and moderate amounts of nickel, zinc, molybdenum, and aluminum are listed as the cation interferences. Anions which interfere are phosphate, sulfate, fluoride, citrate, tartrate, and oxalate, which interfere by competitive complexion.

Both the ascorbic acid concentration and the buffer concentration affect the optical density. The optical density is reduced by an increase in the buffer-ascorbic acid ratio; however, the presence of the buffer is essential to color stabilization. Pyridine also influences the color system, evidently by shifting the absorption spectrum toward longer wave lengths. Without pyridine, the optical density may be determined at 335 or 400 mμ but with pyridine, the measurement should be made at 410 to 440 mμ. Standard practice in this laboratory is to use a measurement wave length of 410 mμ.

Since the majority of samples which fall in the colorimetric range (i.e., 100 to 2000 ppm) are badly contaminated with many of the interferences listed previously, pretreatment of the samples is essential for reliable results. Most of the interferences may be removed by continuous diethyl ether extraction in the presence of calcium nitrate, but such substances as halogens, large amounts of sulfate, and phosphate must be removed before extraction is attempted; ammonia precipitations and perchloric acid fuming usually suffice. Ammonia precipitation also will remove chromium if it is completely oxidized to Cr(VI) because of the solubility of $NH_4$ chromate or dichromate in alkaline solutions. Organic compounds and radicals may be removed by fuming first with nitric acid followed by fuming in nitric and perchloric acid mixture. Both fumings are needed to destroy organics.

After a sample has been processed to remove initial interferences, it is taken up and diluted to volume with 5 N nitric acid. Although the acid concentration is not critical, it should

be such that the extraction mixture is between 3 and 5 N, since best extractions are obtained in this range. The extraction should be performed at room temperature because elevated temperatures affect the distribution coefficient adversely.

The calculation of the uranium content of a sample is made by means of a standard curve, or a linear factor.

## REAGENTS

Ammonium Hydroxide, concentrated
Ascorbic Acid, 12% solution
Buffer Solution (pyridine, acetic acid, ammonium acetate)
Calcium Nitrate, cp crystals
Diethyl Ether
Hydrochloric Acid, concentrated

## EQUIPMENT

Beaker, 100 mls
Continuous Extraction Apparatus (see Figure 4 Page 17)
Cuvettes, to match spectrophotometer or colorimeter
Graduate, 50 mls
Pipette, 5 mls and 10 mls
ph Meter
Spectrophotometer or Colorimeter, capable of reading at 410 mμ

## PREPARATION AND STANDARDIZATION OF SOLUTIONS

### Ascorbic Acid Solution

Compounds - Ascorbic acid - 500 g; distilled water - 4 liters.

The acid is dissolved in the water and mixed thoroughly. Then the mixture is decolorized with activated charcoal and filtered. The reagent is stored in a refrigerator.

### Pyridine Buffer Solution

Compounds - Acetic acid (glacial) - 240 mls; ammonium acetate - 960 g; pyridine (decolorized) - 480 mls, distilled water - 10.7 liters.

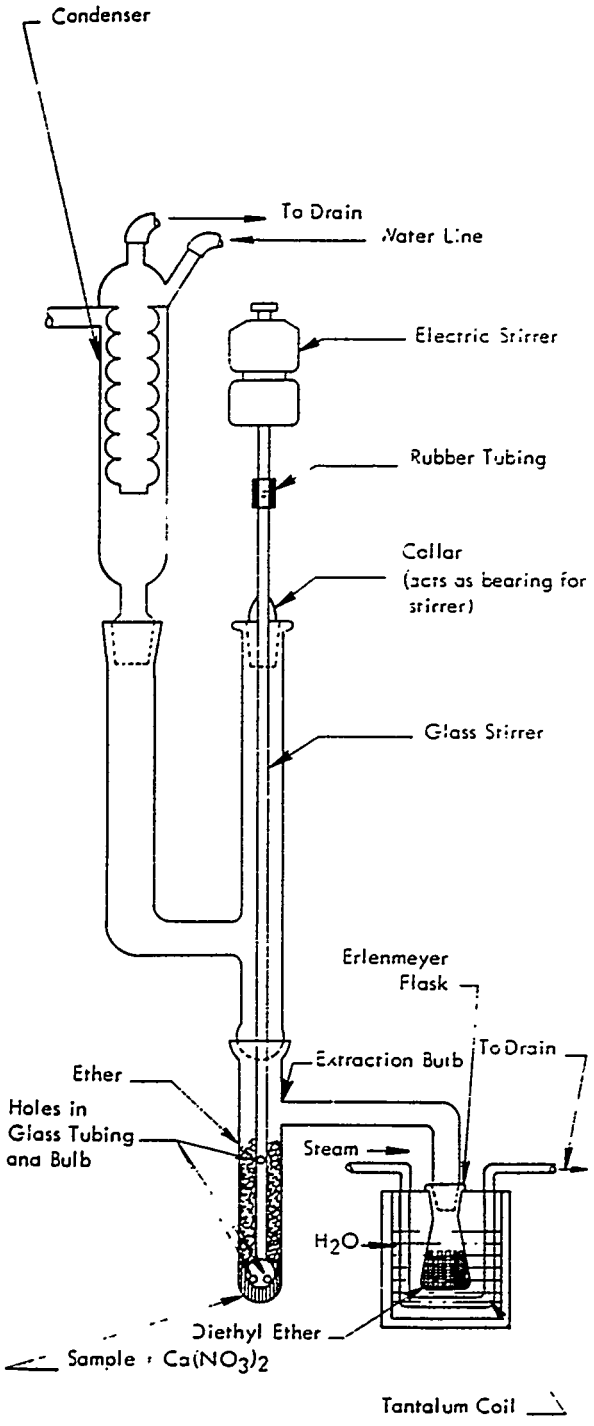



**Figure 4. LABORATORY APPARATUS FOR DIETHYL ETHER EXTRACTION.**

The ammonium acetate is dissolved in several liters of water. This solution is added to the remainder of the water in which the acetic acid and pyridine already have been dissolved. The entire solution is mixed by bubbling air through it.

## Determination of Factor

A new factor must be determined at least once every 24 hours to allow for decomposition of the ascorbic acid solution.

Standard solutions prepared to contain 600, 800, and 1000 µg U/ml are used to determine the factor. Duplicate 5-ml portions of each standard solution are treated in the same manner as the samples.

The factor for each individual solution is then calculated by the equation:

$$F = \frac{\text{ml used} \times \mu\text{g U/ml}}{\text{OD} - \text{blank}}$$

The final factor is an average of the six individual calculations.

# PROCEDURE

## Liquid Sample Preparation

The types of uranium-bearing liquid samples usually received in this laboratory are: water wash, nitric solutions, column water wash, leach solutions, and salvage solutions. These samples should always be checked for sulfates, chlorides, and other interfering elements.

When these samples are received, the uranium concentration is estimated on the

gamma ray counter. The uranium estimation in ppm is recorded on the reverse side of the master requisition card.

A convenient chart to use in preparing the aliquants is given below along with special instructions for certain types of samples.

| Range (ppm) | Aliquant Weight (grams) | Volume (mls) |
|---|---|---|
| 50 - 150 | 30 | 25 |
| 100 - 250 | 30 - 40 | 50 |
| 300 - 500 | 20 - 30 | 50 |
| 500 - 1000 | 8 - 16 | 50 |
| 3000 - 6000 | 2 - 3 | 50 |
| 10,000 - 50,000 | 1 - 2 | 100 |

Samples That Do Not Contain Interfering Cations or Anions - Two appropriate aliquants are weighed in 50-ml tared beakers and the exact weight obtained, then each aliquant is transferred to a 250-ml beaker.

Fifteen to 25 mls of concentrated $HNO_3$ is added and the samples are boiled on a hot plate to destroy any organics that may be present. If a reaction occurs or fumes evolve the boiling is continued until the reaction stops or all fumes are evolved.

The samples are cooled and then transferred to appropriate volumetric flasks and diluted to volume. The samples are ready for the colorimetric determination.

Liquid Samples that Contain Sulfates or Chlorides - Two aliquants are weighed in 50-ml beakers, then each aliquant is transferred to a 250-ml beaker.

Ten mls of concentrated $HNO_3$ is added and the samples are boiled to remove any organics that may be present.

Subsequently, 50 to 75 mls of water is added and the uranium is precipitated with concentrated $NH_4OH$. The precipitate is filtered and washed thoroughly with 1% $NH_4OH$.

The wash is tested for sulfates and chlorides. If any is found the precipitation is repeated. However, if no sulfate or chloride is found, the precipitate is dissolved through the filter paper with hot 5 N $HNO_3$ and then diluted to the desired volume. The samples are ready for the colorimetric determination.

## Solid Sample Preparation

Some of the solid sample types are: muffled solids, paper ash, slag solids, and activated alumina.

The entire sample is ground until it will pass through a 48-mesh sieve and rolled until homogeneous.

Two 3-gram aliquants are weighed and transferred to 250-ml platinum dishes. Samples containing carbon are ignited for one hour at 800 - 900° C.

Twelve to 15 grams of sodium pyrosulfate is added and mixed thoroughly with the sample. The mixture is fused for 30 minutes at 750 - 800° C.

When the crucible is cool, the fusion cake is transferred with hot water to an 800-ml beaker. The beaker is placed in a steam bath to dissolve the fusion cake.

One-half tablespoon of ammonium persulfate crystals is added as well as a few crystals of silver nitrate. The silver nitrate acts as a catalyst and the chromium is oxidized to $Cr^{+6}$ which is soluble in ammonium hydroxide.

The insolubles are filtered off using No. 40 Whatman filter paper. At least six washes are made with hot water. The insolubles are salvaged in the appropriate recycle container.

The uranium is precipitated with concentrated $NH_4OH$. The precipitate is filtered using No. 40 Whatman filter paper and washed with 1% $NH_4OH$. The filtrate is salvaged according to recycle.

Subsequently, the precipitate is dissolved into the original beakers with 5 N $HNO_3$.

The $NH_4OH$ precipitation step is repeated until the filtrate is free of sulfate which is checked with barium nitrate.

The $HNO_3$ dissolution step is repeated. The volume of the sample solution is reduced so that it can be contained in a 100-ml flask. The sample is ready for the colorimetric determination.

## Slurry Sample Preparation

Some of the slurry sample types are: filter press residue, paper ash residue, and reduction residue.

## COLORIMETRIC WORK CARD

| 1951 | 10-18-57 | L-58 | 13-06-88 | g U/g | 123456 |
|---|---|---|---|---|---|
| RECYCLE | DATE | CONT. NO. | BATCH NUMBER | DETERMINATION | REQUISITION NUMBER |

| | | | | | | |
|---|---|---|---|---|---|---|
| ALIQ. NO. | | 1 | 2 | Original Weight | G. 752 | By |
| ALIQ. WT. | G | | | | T. 293 | By DEF |
| | T | | | | N. 459 | |
| BY ABC | N | 3.0019 g | 3.0178 g | Evap. Dish Number 123456 | G. 174 | By |
| DILUTION | Vol. of Flask | 100-ml | 100-ml | | T. 419 | By HJK |
| BY ABC | ml. Pipetted | 5 | 5 | | N. 55 | |
| Reading or Weight | Reading G | .128 | .129 | Jar Number 123456 | G. 175 | By |
| | Blank T | .008 | .008 | | T. 120 | By |
| By EFG | Net Reading N | .120 | .121 | | N. 55 | |
| | | | | Fusion Type $SO_4$ | | |
| | | | | Fusion By CD | Precipitation By MLN | |

CALCULATIONS BY XYZ  Drying Factor = .1198

No. 1: $\frac{.120 \times .00852 \times 20}{3.0019}$ = .0068117 g U/g Dry; .0008160 g U/g Wet

REPORT: Avg. g U/g Dry = .006830

No. 2: $\frac{.121 \times .00852 \times 20}{3.0178}$ = .0068483 g U/g Dry; .0008204 g U/g Wet

Avg. g U/g Wet = .000818

CHECKED BY BF

Y-1570(6-57)

Gram Uranium/Gram Sample (Wet)

g U/g sample (wet) = g U/g sample (dry) X drying factor

0.0008204 = 0.0068483 X .1198

Figure 6. WORK CARD SHOWING TYPICAL ANALYSIS DATA FOR THE COLORIMETRIC DETERMINATION OF URANIUM IN A SLURRY SAMPLE.

## PROCEDURE III - ISOTOPIC ANALYSIS OF $U_3O_8$ USING THE OPTICAL SPECTROGRAPH

### INTRODUCTION

The optical emission spectrograph offers a rapid method for determining the U-235 content of $U_3O_8$ samples. The 4244.3 A uranium emission line pair is due to the isotopes U-238 and U-235. The longer wave length line is due to U-238 while the shorter wave length line is due to U-235. The percents transmission of the U-235 and U-238 emission lines are measured with a Jarrell-Ash No. 203 recording microphotometer. In the region 4244 A to 4245 A four uranium lines are enclosed each of which represents an isotope of uranium and the sum of the intensities of these lines is proportional to the total intensity of uranium. The resolution of the J. A. Spectrograph used in the laboratory is such that in second order only the U-235 and U-238 lines are resolved. The percent U-235 is determined from the ratio of U-235 intensity divided by U-235 intensity plus U-238 intensity multiplied by one hundred. A correction is made for U-234 and U-236 since these lines are not resolved away from the U-235 line.

The precision of the analysis for a sample containing 10 to 90% U-235 is ± 1.0% absolute.

### REAGENTS

Dark Room Supplies (developer, Eastman Kodak D-19; fixer, Eastman Kodak; water, distilled)

### EQUIPMENT

Dark Room (with 3-tank developer, wash tank, and drier)
Dish, platinum, 150 mls
Electrodes, graphite, upper or counter electrode and lower electrode or crater piece
Electrode Holder Block
Filter, blue
Forceps
Microphotometer
Pans, balance - tantalum
Photographic Plate (Eastman, 1 Type SA-2)
Spatula, tantalum
Screwdriver
Spectrograph, Jarrell-Ash

## PROCEDURE

### Preparation of Standards

Standards are prepared to cover appropriate ranges of impurities that are to be determined.

### Sample Preparation

The $U_3O_8$ sample is received in a numbered plastic vial.

The oxide is crushed and mixed in the vial, using a tantalum spatula.

### Charging the Crater Piece

Duplicate 3 1/2-mm x 2-mm crater piece electrodes for each sample are placed in the oblong electrode holder block.

Exactly 10 mgs of the $U_3O_8$ sample is weighed directly onto the tantalum balance pan. Duplicate aliquants are weighed from each sample.

Using forceps, the angled edge of the balance pan is brought over the crater lip of the electrode selected for that aliquant; then by carefully tapping the forceps with the edge of the spatula, the charge is emptied into the electrode.

Each electrode is grasped with the forceps and the bottom of the electrode is tapped against the block to seat the charge firmly.

### Inserting the Photographic Plate

One SA-2 photographic plate is inserted in the center of the 3-plate camera for the Jarrell-Ash instrument.

The camera is centered at 8300 A.

### Arcing and Instrumentation

The spectrograph is prepared for operation.

The required number of counter electrodes (one for each sample) is placed in a 150-ml platinum dish. The dish is placed at the arc stand.

A fresh counter electrode is placed in the upper clip and the first charged crater electrode is placed in the lower clip.

The electrodes are lined up. The iris diaphragm is opened; a blue filter is placed over the diaphragm; the slit is left at ten microns; and the analytical gap is adjusted to 4 mm. The power source is adjusted to 10 amperes DC and the slit timer is set for 70 seconds.

The arc is ignited and the plate is exposed until the power is interrupted by the 70-second timer.

### Plate Processing

The exposed plates are processed by the standard developing method used by the laboratories.

### Plate Reading

The dried SA-2 plate is compared to standard curves determined by a microphotometer. The 4244.3 A U-235 line and the U-238 line are mechanically scanned and the respective percent transmissions of light are obtained.

The percent transmission values are then compared with a standard emulsion calibration curve to determine the intensity values.

## CALCULATIONS

### Percent U-235

The U-235 intensity value and the U-238 intensity value are added.

Formula for g U-235/g $U_3O_8$:

$$\text{g U-235/g} = \frac{\text{U-235 intensity}}{\text{U-235 intensity + U-238 intensity}}$$

Formula for % U-235:

$$\text{\% U-235} = \frac{\text{U-235 intensity}}{\text{U-235 intensity + U-238 intensity}} \times 100$$

# PROCEDURE IV - HIGH PRECISION DETERMINATION OF URANIUM BY POTENTIOMETRIC TITRATION WITH POTASSIUM DICHROMATE

## INTRODUCTION

Since this method is designed for the determination of uranium with extremely high precision, more knowledge of a sample is required than for other less precise methods. The uranium content of the sample must be known to ± 0.1%, and the "pretitrational" history of the sample must be known to be sure that nitrate ion and other interferences such as iron are not present. Samples titrated must contain enough uranium to realize the desired precision; a sample containing 2 grams of uranium will have a limit of error (95%) of ± 0.06%, and a 5-gram sample will have an experimental limit of error of ± 0.03%.

The determination consists of: (1) preparing the sample for analysis by dissolving in nitrate-free 7.5% sulfuric acid solution, (2) passing the solution through a 2% zinc amalgam to reduce the uranium to a mixture of U(IV) and U(III), (3) aeration of the reduced sample to the U(IV) potential break, (4) adding a predetermined, accurately weighed amount of standard potassium dichromate, and (5) finishing the titration by either forward titration with ferric ammonium sulfate and standard dichromate solution, or by backward titration with ferrous ammonium sulfate. A slight excess of solid dichromate is added following aeration if backward titration is employed, and a slight deficiency of solid dichromate is added if forward titration is employed.

In the forward titration, most of the uranium is oxidized to U(VI) by the dichromate, and the slight excess of U(IV) remaining is oxidized to U(VI) by ferric iron. The ferrous iron thus formed is titrated back to ferric by standard dichromate solution. In the backward titration, a slight excess of dichromate is added, the excess being determined by the volume of standard ferrous solution necessary to reduce the dichromate. The forward titration is not quite as easy to handle in the laboratory as the backward titration, but does not suffer from the difficulty that an unstable solution is used for titration. In either the forward or backward titration, the second end point is that of ferrous-dichromate couple and not that of uranium as in other titration methods. This laboratory uses the backward titration method.

Iron is a major interference in both titrations. Fifty micrograms of iron is equivalent to approximately 0.01% uranium. Therefore, in the general run of samples, it is necessary to correct for the iron content either from spectrographic iron results or from a colorimetric determination of iron, since the iron will titrate along with the uranium. Other elements, such as molybdenum, which are reduced in the Jones Reductor and oxidized by dichromate will yield high results, but iron is the most commonly occurring interference in significant quantities. A few other elements, such as copper, will plate out in the reductor and slowly poison it.

The presence of excessive amounts of iron also interferes by an oxidation-reduction cycle during the aeration step. Ferrous iron will be oxidized by air to Fe(III), which will oxidize U(IV) to U(VI), the Fe(III) in turn being reduced to Fe(II), and so on around again. This effect will not be serious if the aeration step is stopped immediately at the equivalence point and the air replaced by nitrogen.

The complete data for a "high precision" analysis is recorded on an IBM mark sense card using an IBM Electrographic pencil. The IBM cards are transferred to the Tabulating Services Department for calculation.

## REAGENTS

Ferrous Ammonium Sulfate Solution, ~ 0.05 N in 7.5% $H_2SO_4$
Perchloric Acid (concentrated 70%)

Potassium Dichromate (cp crystals, primary standard dried at 110° C for 1 hr and stored in an efficient desiccator)
Sulfuric Acid, 7.5% by volume
Zinc Amalgam, ~ 2%

## EQUIPMENT

Burettes, 10-ml Normax and 100-ml MCA 3
Entrainment Trap, see Figure 7, Page 31
Fisher Filtrator with 13 1/4 in x 4 3/8 in Diameter Filtering Mantle
Flasks, 500-ml wide mouth Erlenmeyer, 125-ml and 250-ml Erlenmeyer
Gas Scrubbing Tower
Jones Reductor, 15 mm ID, Figure 7, Page 31
Potentiometer, a vacuum tube voltmeter in connection with platinum-calomel electrode system

## PREPARATION AND STANDARDIZATION OF SOLUTIONS

### Ferrous Ammonium Sulfate Solution

Compounds - $Fe(SO_4)\cdot(NH_4)_2SO_4\cdot 6H_2O$ (cp crystals) - 160 g; $H_2SO_4$ (concentrated) - 80 ml; distilled water - 2 liters.

The ferrous ammonium sulfate solution is prepared by adding 80 mls of concentrated sulfuric acid to 2000 mls of distilled water. When the mixture is cool, 160 grams of ferrous ammonium sulfate is added and then diluted with distilled water to eight liters in a carboy. The solution is stirred until thoroughly mixed.

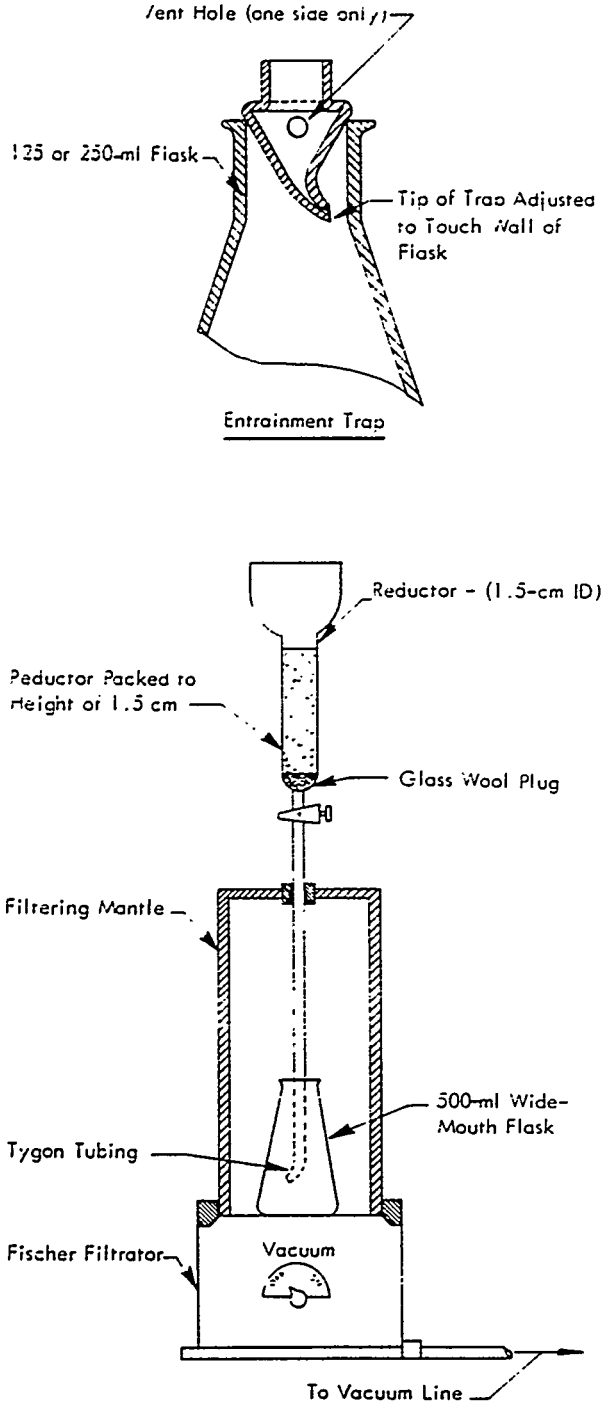



**Figure 7. ENTRAINMENT TRAP AND JONES REDUCTOR ARRANGEMENT.**

## Standardization of Ferrous Ammonium Sulfate Solution

The ferrous ammonium sulfate solution is standardized against primary standard potassium dichromate.

One-tenth gram of primary standard potassium dichromate is accurately weighed and quantitatively transferred to a 500-ml Erlenmeyer flask. The salt is dissolved in 100 to 150 mls of 7.5% sulfuric acid.

The Erlenmeyer flask containing the dissolved potassium dichromate is placed on the titration assembly. With a 100-ml burette, ferrous ammonium sulfate solution is added to the dichromate solution until the equivalence point is reached. The potential break at the equivalence point should be on the order of 200 mv/drop.

The ferrous ammonium sulfate solution is standardized daily since the solution is not protected by $CO_2$.

Formula for grams/ml equivalent:

$$Fe^{+2} \text{ equivalent} = \frac{\text{grams } K_2Cr_2O_7 \text{ used}}{\text{ml of titer used}}$$

## Preparation of Zinc Amalgam

Compounds - Zinc Metal (30-mesh) - 180 grams; Mercuric Chloride - 2 grams; HCl (concentrated) - 2 mls; distilled water 500 mls.

The zinc amalgam is prepared by adding 2 mls of concentrated hydrochloric acid to 500 mls of distilled water, then adding 180 grams of zinc metal, 30-mesh, and finally 2 grams of mercuric chloride.

The solution is stirred well and the zinc is agitated vigorously so that the entire surface is exposed. Subsequently, the acid solution is drained off and the zinc is rinsed with several portions of distilled water.

## PREPARATION OF JONES REDUCTOR

### Packing Jones Reductor

After the reductor has been cleaned thoroughly, a layer of glass wool is packed into the bottom. The prepared zinc amalgam is poured on top of the packing until the narrow part of the reductor is about three-fourths filled; then the amalgam is worked into a solid packing without channels or air pockets. The packing is washed with 7.5% sulfuric acid and then rinsed with water.

A newly packed reductor must be appropriately "conditioned" before an "unknown" sample is passed through in order to eliminate the possibility of a low result. Consequently, a 5-gram uranium standard sample is passed through the reductor by several washings with 7.5% sulfuric acid.

Best service is obtained from the reductor if the amalgam is kept under water at all times. After a long inactive period, alternate rinses with distilled water and 7.5% sulfuric acid seem to increase the activity of the amalgam.

## PROCEDURE

### Sample Preparation

Duplicate aliquants of a sample, each of which contain 5 grams of uranium in which the uranium concentration is known to ± 0.1%, are prepared for analysis. The limit of error on a 5-gram sample is approximately ± 0.03%.

Uranium Metal - The metal is dissolved in a 1:4 solution of hydrochloric acid; then 5 mls of perchloric acid is added and the solution is fumed to near dryness. Subsequently, the aliquant is transferred to the Jones Reductor by several washings with 7.5% sulfuric acid.

Uranium Oxides - The oxides are dissolved directly in approximately 15 mls of perchloric acid. The aliquants are fumed to near dryness and then transferred to the Jones Reductor with 7.5% sulfuric acid.

Uranium Tetrafluoride - $UF_4$ may be prepared for titration by either of two methods: (1) The $UF_4$ is pyrohydrolyzed to $U_3O_8$; subsequently, the oxide is dissolved in approximately 15 mls of perchloric acid. The aliquants are fumed to near dryness and then transferred to the Jones

Reductor with 7.5% sulfuric acid. (2) The $UF_4$ aliquants are dissolved in a 10% solution of ammonium oxalate and the oxalate is destroyed by boiling with sulfuric acid. The solution is adjusted to 7.5% sulfuric acid and the reduction is carried out.

Uranium Liquids - Aliquants of sample equivalent to 5 grams of uranium are placed in flasks and fumed free of nitrates and chlorides with 15 mls of perchloric acid. Usually three fumings are necessary to get rid of all nitrates.

Reduction

A 500-ml wide mouth Erlenmeyer flask containing a magnetic stirrer is placed under the delivery tube of the Jones Reductor in the Fisher Filtrator, and a slight vacuum is applied (see Figure 7, Page 31).

The uranyl perchlorate solution is transferred quantitatively with 10 to 20 mls of $H_2SO_4$ to the top of the column; the solution is allowed to run through the column at a flow rate of 75 - 80 mls/minute until the level of the solution is just above the top of the column packing; and then another portion of 7.5% $H_2SO_4$, which has been used to rinse the sample flask, is added. This operation is repeated until the sample flask has been rinsed into the column 3 or 4 times.

Washing the top reservoir of the column with 7.5% $H_2SO_4$ is continued until a total of 150 to 200 mls of solution has been collected in the receiver flask.

Aeration

The 500-ml Erlenmeyer flask is removed from the reductor, and it is placed into position in the titration assembly (see Figure 2, Page 12).

Sulfuric acid-washed air is passed through the sample solution at a brisk rate not vigorous enough to cause spattering, while at the same time stirring is maintained at full speed with the magnetic stirrer. This will ensure the shortest possible oxidation time, since the rate of oxidation is increased with increased stirring speeds.

The electronic millivoltmeter is adjusted to read negative potentials; the potential should be about -200 mv or less. The potential will remain fairly constant until most of the U(III) has been oxidized to U(IV).

When the potential has risen to about -100 mv, the air stream is replaced with oxygen-free nitrogen. In the process of changing the gas, the potential should rise past zero to some positive potential; consequently, it will be necessary to reverse the polarity of the electrodes.

Sometimes the final potential will be as high as +300 mv. At this stage the solution should be a light green.

At this point the forward or backward titration may be employed with equal effectiveness. Since this laboratory uses only the backward titration, the forward titration will not be discussed in detail.

Titration

Within a minute of the first potential break, the weight of potassium dichromate calculated from the following formula is added to the sample solution.

$$g\ K_2Cr_2O_7 = \frac{(g\ U\ taken)\ (equiv\ wt\ K_2Cr_2O_7)}{equiv\ wt\ of\ U} + 0.002\ g$$

The tolerable deviation from this weight is +0.001 to 0.000 g.

Subsequently, the accurately weighed potassium dichromate aliquant is carefully transferred to the titration flask by several washings with distilled water. The solution is then stirred vigorously for at least five minutes, or two minutes after the dichromate has dissolved completely. The potential of the solution at this stage should be +650 mv or higher, indicating an excess of dichromate.

Standardized ferrous ammonium sulfate is added dropwise from a 10-ml Normax burette and the potential change is observed after the addition of each drop. When the end point is approached, as indicated by slight dipping of the galvanometer needle, the titration assembly is rinsed with water.

The titer is added continuously until the end point is passed. The potential break at the end point should be about 50 to 150 mv/drop of titer. The volume of titer including the drop causing maximum potential change is recorded.

When the titration is completed, the sample solution in the flask is salvaged in the appropriate recycle salvage container.

## CALCULATIONS

### Grams of Uranium per Gram of Sample

Formula 1 -

$$g\ excess\ K_2Cr_2O_7 = ml\ Fe^{+2} \times g/ml\ equiv$$

Formula 2 -

$$\text{actual wt } K_2Cr_2O_7 = \text{total wt } K_2Cr_2O_7 - \text{excess } K_2Cr_2O_7$$

Formula 3 -

$$\text{factor} = \frac{\text{equiv wt of U}}{\text{equiv wt of } K_2Cr_2O_7}$$

Formula 4 -

$$\text{g U/g sample} = \frac{\text{actual wt } K_2Cr_2O_7 \times \text{factor}}{\text{grams of sample}}$$

$$\text{corrected g U/g sample} = \text{g U/g sample} \times \text{analyst's factor}^{(a)}$$

Iron Corrections

For precise analysis of the grams uranium/gram sample, an iron correction is made. This correction is calculated from the spectrographic iron results or from a colorimetric determination of iron.

The iron correction from a colorimetric determination is determined by the following formulas:

Formula 1 -

$$\text{Fe correction} = \text{g Fe/g sample} \times \text{Fe equiv of U}$$

Formula 2 -

$$\text{corrected g U/g sample} = \text{g U/g sample} - \text{Fe correction}$$

---

(a) The analyst's factor is a correction factor which is used to compensate for the individual analyst bias on analyses by this "high precision" titration method. The correction factor is statistically determined for each analyst from their analyses of a control standard:

$$\text{Analyst's factor} = \frac{\text{Overall mean of the control standard}}{\text{Individual analyst mean of the control standard}}$$

Since the spectrographic iron result is expressed in parts per million of iron in uranium oxide, the iron equivalent of uranium must be determined in terms of uranium metal.

Briefly, the formulas for iron corrections from spectrographic iron results are:

Formula 1 -

$$1 \text{ ppm} = 0.000001 \text{ g/g}$$

Formula 2 -

$$\text{Fe correction} = \frac{\text{g U, g sample} \times \text{g Fe/g } U_3O_8}{0.40}$$

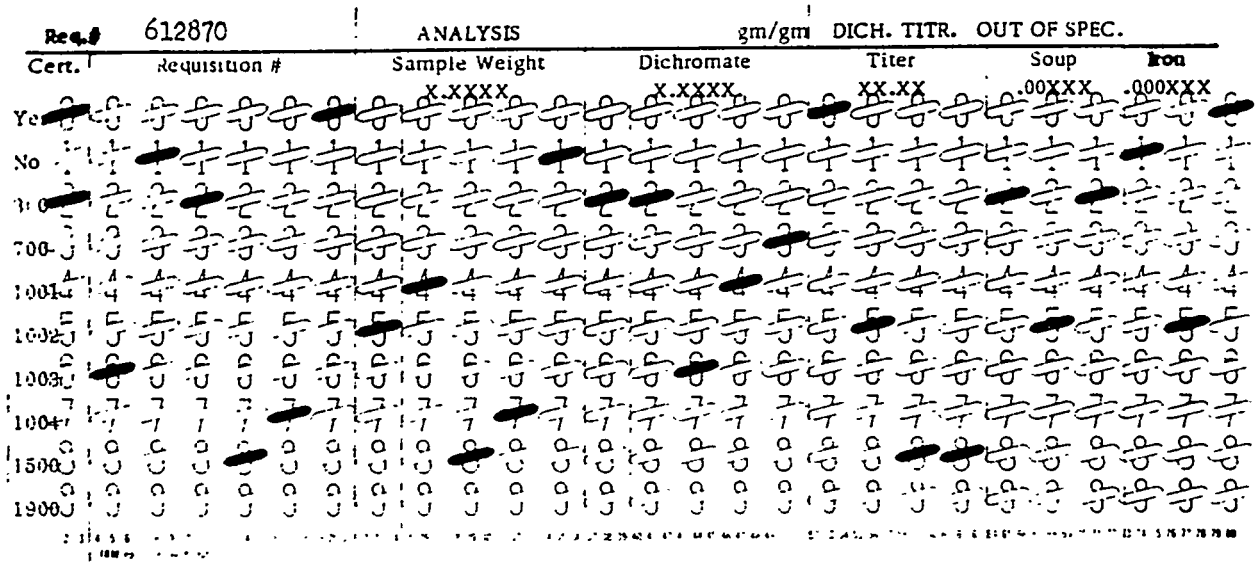
Formula 3 -

$$\text{corrected g U/g sample} = \text{uncorrected g U/g sample} - \text{Fe correction}$$

The complete calculations are checked to ascertain the accuracy.

SAMPLE WORK CARD

A typical determination of the grams of uranium/gram of sample recorded on an IBM mark sense card with an IBM Electrographic pencil is shown in Figure 8 (front of card), and Figure 9 (back of card).




Column 1 - The material certification is marked either "yes" or "no" depending upon the purpose for which the material is to be used.

The material type (recycle) is marked (300).

Column 2 - The requisition number of the sample is marked (612870).

Column 3 - The sample weight (aliquant weight) is marked (5.4871 g).

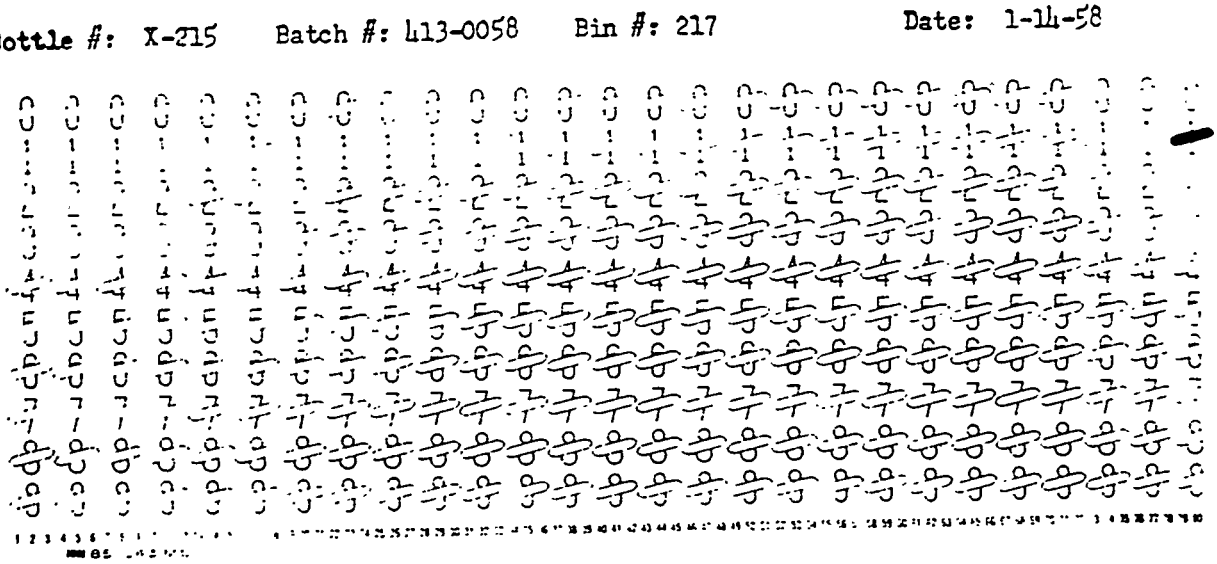
Column 4 - The weight of potassium dichromate used is marked (2.2643 g).

Column 5 - The ml of ferrous ammonium sulfate used is marked (05.88 ml).

Column 6 - The $Fe^{+2}$ equivalent factor (Soup) is marked (0.00252).

Column 7 - The g Fe/g sample is marked (0.000150 g Fe/g).

**Figure 8. IBM MARK SENSE CARD USED TO RECORD GRAMS-OF-URANIUM-PER-GRAM-OF-SAMPLE DATA. (Front Side of Card)**




The analyst's code number is marked in the last space on the card reading from left to right.

Figure 9. IBM MARK SENSE CARD USED TO RECORD GRAMS-OF-URANIUM-PER-GRAM-OF-SAMPLE DATA. (Reverse Side of Card)

## PROCEDURE V - FLUORIMETRIC DETERMINATION OF LOW CONCENTRATIONS OF URANIUM BY EXTRACTION AND DILUTION METHODS

### INTRODUCTION

The fluorimetric method is used for the determination of microgram and sub-microgram quantities of uranium in liquid and solid samples. The method depends upon the fact that certain uranium compounds, presumably the uranyl structure will fluoresce if illuminated with ultraviolet light. The fluorescence of uranium is most intense if the uranium is fused into a matrix of sodium fluoride. When other factors are controlled, the intensity of the fluorescence, from a phosphor of uranium fused in sodium fluoride, is a linear function of uranium concentration.

The fluorimetric procedure consists of four steps: (1) aliquanting the sample by weighing or by pipetting followed by a specific gravity measurement, (2) extracting or diluting to eliminate quenching, (3) drying and fusion of the sample in sodium fluoride-2% lithium fluoride flux, and (4) measuring the fluorescence and calculating the uranium content.

Certain foreign elements, such as iron, chromium, copper, nickel, zinc, and zirconium, cause "quenching", which is a reduction in the fluorescence intensity for a specific amount of uranium. The degree or amount of quenching is dependent on the concentration of impurities in the sodium fluoride flux and is independent of the uranium concentration. Thus quenching can be decreased below detectable limits by dilution or by separation of the uranium from impurities by an extraction with tri-n-octyl phosphine oxide in varsol. Due to the high affinity of tri-n-octyl phosphine oxide (TOPO) for the uranyl ion, the extraction is essentially independent of the nature of the sample solution in which the uranium is contained. This makes the extraction method an ideal choice for the fluorimetric analysis of uranium from miscellaneous plant solutions which may contain many unknown impurities.

The range of the fluorimetric analysis is limited by several factors. Above a certain uranium to sodium fluoride flux weight ratio, the uranium begins to quench its own fluorescence and the relation between the fluorescence intensity and the uranium concentration ceases to be linear. The amount of sodium fluoride flux used for each fusion is limited by a practical size platinum dish and the heating area needed for the fusion. A platinum dish which will hold a 0.2-gram pellet of flux meets these conditions. This weight flux pellet will absorb a 100-lambda volume of liquid which is about the smallest volume capable of being measured with the necessary accuracy and speed for routine analyses. Samples which contain uranium concentrations of 0.01 µg/ml to 50.0 µg/ml after dilution or extraction can be analyzed by this procedure. A 100-lambda portion would represent 0.001 µg to 5.0 µg of uranium fused into the 0.2-gram pellet of flux. For amounts greater than 5.0 µg, dilutions are made for maximum accuracy and precision.

A 2% mixture of lithium fluoride with sodium fluoride is used as a flux because the mixture

is more easily dissolved from the platinum dishes and yields greater precision than pure sodium fluoride without a detectable decrease in the fluorescence intensity. Since sodium fluoride with 2% lithium fluoride is a very corrosive flux, only platinum can be used to contain it and even platinum is attacked if the fusion is performed in an oxidizing atmosphere. To meet the requirements of temperature, atmosphere, and maximum number of samples capable of fusion at one time, a Fletcher burner modified for use with compressed air is used.

For the extraction technique, the limit of error for one analysis in the range of less than one part per million uranium is approximately ± 50% while the limit of error for one analysis in the range of one to one hundred parts per million uranium is approximately ± 20%.

## REAGENTS

Acetone
Aluminum Nitrate (monohydrate)
Carbon Tetrachloride
Chloroform
Distilled Water
Lithium Fluoride
Methyl Alcohol
Nitric Acid (concentrated)
Perchloric Acid (concentrated)
Sodium Fluoride (Baker and Adamson only)
Sulfuric Acid (concentrated)
Tri-n-octyl Phosphine Oxide
Varsol

## EQUIPMENT

Beakers, 150 mls, 400 mls, and 800 mls
Burner, modified Fletcher
Crucibles, porcelain, No. 3 (tall form)
Cylinders, graduated, 50 mls and 100 mls
Dishes, platinum-evaporating, 200 mls
platinum-fusion, fabricated by forming in a custom made punch and die from 20-mil and 25-mil platinum sheet, 11/16-inch diameter, 1/16-inch lip width, and 1/8-inch cup depth
Filter Paper (S and S), 12.5 cm
Filter Pulp
Flasks, volumetric, 100 mls, 1000 mls, and 2000 mls
Forceps
Hot Plate

Hydrometers
Infra-radiator Lamps and Stand
Jars, flux-dispensing, (glass, wide mouth), 2 7/8 inches diameter and 3 1/2 inches long with plastic screw cap
flux-storage, 5-pound amber glass, wide mouth, reagent
Pelletizer, delivering a 0.2-gram pellet
Pipettes (Normax), 1 ml, 2 mls, 5 mls, and 10 mls; calibrated, 1 ml and 10 mls; 100-lambda and pipette syringe
Pliers, long-handled
Shaker, mechanical
Tongs, transfer
Torch, propane-oxygen
Trays (Lavite), 12 3/8 inches long, 3 7/8 inches wide, with 40 wells (10 rows with 4 in each row) spaced 1 inch apart on centers
Vials, extraction and dilution (glass), 20 mls, 2 1/4 inches long, 1 inch diameter with plastic screw cap
Watch Glasses, 4 1/2 inches
Wire Screen (Nichrome), 5-inch diameter circle cut from 16-mesh, 5 gauge stock

## PREPARATION OF REAGENTS

### Sodium Fluoride-2% Lithium Fluoride Flux

Compounds - NaF (Baker and Adamson only) - 3 lbs; LiF - 28 g.

The contents of three one-pound jars of Baker and Adamson sodium fluoride are emptied into a clean, 5-pound amber glass, reagent jar and a 28-gram portion of lithium fluoride is added. The jar is then placed on the mechanical roller and the contents rolled for 48 hours.

### Tri-n-octyl Phosphine Oxide and Varsol Solution

Compounds - TOPO - 100 g; Varsol - 5 liters.

The 100 grams of tri-n-octyl phosphine oxide is dissolved in 1000 mls of varsol by heating in a steam bath. The solution is then transferred to a 5-pound amber glass, reagent jar and sufficient varsol is added to make a total volume of five liters. The solution is mixed well. This gives an approximate 0.05 molar TOPO-varsol solution.

### Aluminum Nitrate Solution

Compounds - $Al(NO_3)_3 \cdot 9H_2O$ - 375 g; $HNO_3$ (concentrated) - 64 mls; distilled water - ~ 936 mls.

The aluminum nitrate solution is prepared by: (1) dissolving 375 grams of aluminum nitrate in 500 mls of distilled water, (2) adding 64 mls of concentrated nitric acid, and (3) diluting to 1000 mls with distilled water. The solution is stored in a one liter reagent bottle.

## CLEANING EQUIPMENT

### Cleaning the Platinum Fusion Dishes

The platinum fusion dishes containing the sample pellets are placed in a 200-ml platinum evaporating dish and sufficient distilled water is added to moisten all the pellets. The fusion dishes must not be placed one on another or overlap in the evaporating dish.

Sufficient concentrated sulfuric acid is added to cover all the fusion dishes, after which the evaporating dish is placed on a hot plate and the fusion dishes are boiled until the sample pellets dissolve.

The evaporating dish is removed from the hot plate, the acid is allowed to cool after which it is poured off, and water is allowed to run into the dish for approximately two minutes to remove all the acid from the fusion dishes.

After the water is poured from the evaporating dish, sufficient concentrated nitric acid is added to cover the fusion dishes. The evaporating dish is placed on a hot plate and the fusion dishes are allowed to boil in the acid for approximately five minutes. The evaporating dish is removed from the hot plate and distilled water is allowed to run over the fusion dishes for approximately two minutes to remove all the acid.

The preceding step is repeated.

After the fusion dishes are rinsed thoroughly with distilled water, they are transferred for storage to a Pyrex dish filled with distilled water.

Just prior to use, the platinum fusion dishes are removed from the Pyrex dish and the step involving the addition of concentrated $HNO_3$ and boiling is repeated.

The thoroughly rinsed platinum fusion dishes are placed in Lavite trays and the trays are placed under infrared lamps until the fusion dishes are dried. They are now ready for use.

### Cleaning Pipettes

All pipettes are cleaned as soon as possible after use to prevent samples from drying inside them.

Lambda, 1-ml, and 2-ml pipettes are cleaned according to the following procedures:

Used on Aqueous Sample Solutions - (1) Using a vacuum line, the pipettes are rinsed thoroughly with water for approximately five seconds. This rinse is repeated three times. Fresh water is used for each set of approximately 12 pipettes. (2) After the water rinse, the pipettes are rinsed as in Step 1 in the following order: concentrated nitric acid, distilled water, and acetone. (3) They are allowed to dry.

Used on Organic Sample Solutions - Oils, Carbitol, and Perclene - (1) Using a vacuum line, the pipettes are rinsed for approximately five seconds with the organic solvent used to dilute the samples. This rinse is repeated three times. The solvent is discarded after a set of approximately 12 pipettes have been rinsed. (2) After the solvent rinse, the pipettes are rinsed as in Step 1 with acetone, distilled water, concentrated nitric acid, distilled water, and acetone. (3) They are allowed to dry.

All cleaning solutions are changed every 24 hours.

Larger pipettes (those other than lambda, 1-ml, and 2-ml) are cleaned according to the following procedures:

Used on Aqueous Sample Solutions - (1) The pipettes are rinsed with water by holding them upright under the water faucet for approximately one minute. They are allowed to drain completely several times. (2) They are then rinsed with concentrated nitric acid by placing them in plastic cylinders and filling the cylinders with nitric acid. This acid is changed each shift if many pipettes are cleaned. (3) After the nitric acid rinse, the pipettes are rinsed well by holding them upright under the distilled water faucet. (4) The pipettes are dried by pulling acetone or methyl alcohol through them with a vacuum line.

Used on Organic Sample Solutions - (1) The pipettes are rinsed well with the solvent used to dilute the samples. (2) After the solvent rinse, they are rinsed in the following order: acetone, distilled water, concentrated nitric acid, distilled water, and acetone. (3) The pipettes are then dried.

Cleaning Hydrometers and Graduated Cylinders

Used on Aqueous Sample Solutions - The hydrometers and graduated cylinders are cleaned by first rinsing with distilled water several times, then rinsing with methyl alcohol, and finally drying by a compressed air stream.

Used on Organic Sample Solutions - The hydrometers and graduated cylinders are cleaned by first rinsing with an organic solvent that will dissolve the sample material, then rinsing with methyl alcohol, and finally drying by a compressed air stream.

## PROCEDURE

### Estimation of Uranium Concentration in Sample

The fluorimetric method is used for samples containing < 100 ppm uranium on the solution basis and for organics that contain < 1000 ppm uranium on the solution basis.

The parts per million uranium in each sample solution is estimated by reading each sample on the gamma counter; the estimated ppm uranium is recorded on the reverse side of the master card, Request for Analysis, by the personnel receiving the samples.

### Specific Gravity Measurement

Since most samples require a gram-of-uranium-per-gram-of-sample answer, a specific gravity measurement is necessary in order to calculate the uranium on a weight basis.

A 45-ml portion of the sample is transferred into a clean, dry 50-ml graduated cylinder. If a 100-ml graduated cylinder is used, a 90-ml portion of sample is necessary.

A hydrometer with a specific gravity scale of 0.940 - 1.010 is selected.

The hydrometer is immersed in the liquid sample in the graduated cylinder; the scale on the hydrometer must be in an upright position.

If the hydrometer scale sinks below the surface of the liquid, another hydrometer with lower scale readings is selected. If the hydrometer scale should be entirely above the surface of the liquid, a hydrometer with higher scale readings is selected.

The preceding step is continued until a hydrometer is selected which will be suspended in the liquid so that the surface of the liquid will intersect a portion of the graduated specific gravity scale. There is a tendency for the hydrometer to adhere to the sides of the cylinder and thus give a false reading. The hydrometer is tapped several times to make sure that it floats freely before a reading is taken.

The specific gravity measurement is taken at the point where the hydrometer scale and the surface of the liquid intersect. This reading is recorded on the master requisition card for the sample and is used in calculating the gram uranium per gram of sample answer. The specific gravity is used as the gram weight of one milliliter of the sample.

After the specific gravity measurement has been made, the sample is returned to the original sample container. The hydrometers and graduated cylinders are cleaned as previously described (Page 43).

## Sample Preparation for Extraction

Aqueous Liquids - Aqueous liquids such as raffinates, condensates, nitric solutions, feeds, general salvage, ammonia filtrates, or peroxide filtrates generally require no sample preparation and are pipetted directly into the extraction vial.

Copper and Brass Metal Samples - These samples are prepared according to the following procedure:

An approximate 20-g portion of the copper or brass turnings is placed in a clean 400-ml beaker and sufficient 1:3 $HNO_3$ acid solution is added to cover the turnings. The solution is heated if necessary.

When the acid solution begins to turn green, it is poured out and the turnings are rinsed well with distilled water, then with alcohol, and finally with acetone. The metal is dried by a compressed air stream.

The cleaned sample aliquant is weighed to the nearest 0.1 mg and transferred to a clean 400-ml beaker. Fifty mls of distilled water and 50 mls of concentrated $HNO_3$ are added and the beaker is covered with a watch glass.

When the reaction has ceased or the sample is completely in solution, the beaker is placed on a hot plate and the solution is evaporated to less than 100-ml volume.

The solution is transferred from the beaker to a 100-ml volumetric flask, cooled, and diluted to volume with distilled water.

Ten mls of the solution is pipetted into a 20-ml extraction vial, then 2 mls of concentrated $HNO_3$ and 2 mls of TOPO-varsol solution are added. The sample is now ready for extraction, fusion, and measuring the fluorescence (see the description of these procedures on Pages 48 and 50.

Steel Samples - Steel samples are prepared as follows:

The total steel sample received is degreased in the following manner: washed with acetone and then with ether, dried, "pickled" with 1:1 HCl solution, washed with distilled water, washed with acetone, and dried with a slow stream of air.

An approximate 4-g portion of the cleaned steel chips or turnings is weighed to the nearest 0.1 mg into a clean 400-ml beaker. Fifty mls of concentrated $HNO_3$ and 50 mls of reagent-grade concentrated perchloric acid are added and the beaker is covered with a watch glass.

The beaker is placed on a hot plate set at low heat and the sample is heated very carefully until the evolution of hydrogen bubbles and a green color indicate that the reaction has begun. The beaker is removed from the hot plate immediately. The reaction will continue unaided and will become violent if heating is continued. After the reaction slows, gentle heat may be applied again.

If the sample does not react, 10 - 20 mls of concentrated HCl is added slowly. In most cases the sample will go into solution at this point; however, if the sample does not completely dissolve, most of the acid is fumed off.

Any undissolved salt is dissolved in distilled water, heating if necessary. The solution which should not be cloudy or turbid is transferred from the beaker to a 100-ml volumetric flask, cooled, and diluted to volume with distilled water.

Ten mls of the sample solution is pipetted into a 20-ml extraction vial, then five mls of concentrated $HNO_3$ and two mls of TOPO-varsol solution are added. The sample is now ready for the usual extraction (see Page 48) with the following exceptions: (1) After shaking for three minutes, the layers are allowed to separate; the aqueous layer is removed and salvaged. (2) The sample is backwashed with three mls of concentrated $HNO_3$ and 11 mls of distilled water to remove the iron and chromium from the organic layer. (3) The aqueous layer is again removed and the backwash step is repeated.

The sample is now ready for fusion and measuring the fluorescence as described on Pages 49 - 51.

Meltemp Oil Samples - Meltemp samples are prepared by the following procedure:

These oil samples which are a very viscous, high-flash-point oil contain suspended $U_3O_8$ or other solids. A specific gravity measurement is determined by weighing a volume of the oil.

The bottle containing the entire oil sample is weighed to determine the gross weight. All the oil is transferred to an 800-ml beaker, then the bottle is rinsed with methyl chloroform. The bottle is dried and weighed to determine the tare weight. All material must be at room temperature before weighing.

The oil, after being diluted with methyl chloroform until it is thin enough to transfer, is transferred from the beaker to a 1000-ml volumetric flask, diluted to volume with methyl chloroform, and shaken well. (The dilution should be approximately 10 to 1 or 1000 mls of solution for 100 grams of oil.)

Using a Buchner funnel under vacuum, the sample solution is filtered through a pad of filter

pulp covered with 12.5-cm S and S filter paper into a 2-liter receiving flask. The sample is poured carefully into the center of the paper to prevent the solids from escaping around the edges.

After the filtering step is completed, the vacuum is allowed to continue in order to dry the filter paper. The dried filter paper is removed from the funnel to a No 3 tall porcelain crucible and the funnel is rinsed with methyl chloroform into the 2-liter receiving flask.

The filter paper in the crucible is ignited in a muffle furnace at 850° C for 30 minutes.

The ignited solids are leached in hot $HNO_3$. The solids are dissolved and transferred from the crucible to a 100-ml volumetric flask. When cool, the sample is diluted to volume with distilled water.

Five mls of the sample solution in the volumetric flask is pipetted into a 20-ml extraction vial; then five mls of TOPO-varsol solution and five mls of concentrated $HNO_3$ are added. The sample is now ready for extraction, fusion, and measuring the fluorescence (as described on Pages 48 - 51). This gives the gram uranium per gram in the aqueous phase.

All the organic filtrate is transferred from the 2-liter receiving flask to the original 1000-ml volumetric flask. If evaporation has occurred, methyl chloroform is added to bring the volume up to the calibration mark. The filtrate is now ready for fusion and measuring the fluorescence as described on Pages 49 - 51. This gives the gram uranium per gram in the organic phase.

The total gram uranium per gram of Meltemp oil is the sum of the gram uranium per gram in the aqueous phase and the gram uranium per gram in the organic phase. The method of calculation is described on Page 51.

Potassium and Lithium Carbonate Fused Salt Samples - These samples are prepared for extraction by the following procedure:

This type of sample is received in the laboratory in short lengths of steel pipe in which a sample of the salt has solidified.

Any sample material on the outside of the pipe is removed with a wire brush.

With long-handled pliers, the pipe is held over a labeled, porcelain crucible and the salt is melted from the pipe with a propane-oxygen torch.

The entire sample is weighed to the nearest 0.1 mg into a tared beaker, dissolved in 1:1 $HNO_3$ solution, transferred from the beaker to a 250-ml volumetric flask, and diluted to volume with distilled water.

Five mls of the sample solution is pipetted into a 20-ml extraction vial, then five mls of TOPO-varsol solution and five mls of concentrated $HNO_3$ are added. The sample is now ready for extraction, fusion, and measuring the fluorescence, (Pages 48 - 51).

Solid Samples - Solid samples are prepared as described in the section "Colorimetric Determination of Uranium". When the uranium concentration in these samples is too low to be determined colorimetrically, the volumetric flasks containing the uranium solutions are transferred to the fluorimetric area for analysis.

## Sample Preparation for Dilution

Aqueous Samples with Low U Concentration - A 1-ml aliquant of sample is pipetted into a 20-ml dilution vial and nine mls of distilled water is added from a graduated pipette. The vial is capped and the sample is mixed thoroughly. The sample is now ready for fusion and measuring the fluorescence (see Pages 49 - 51).

Aqueous Samples with High U Concentration - A 1-ml aliquant of sample is pipetted into a 100-ml volumetric flask and diluted to volume with distilled water. These samples can usually be identified by their yellow color. The sample is now ready for fusion and measuring the fluorescence (Pages 49 - 51).

Organic Solvent and Light Oil Samples - These samples require the following treatment:

First, it is necessary to find a proper solvent to dilute the sample. The usual procedure is to try the following solvents in the order listed: varsol, carbon tetrachloride, acetone, chloroform, and distilled water. A small quantity of sample is tested with the solvents to determine its miscibility. In most cases varsol is satisfactory.

A 1-ml aliquant of the sample is pipetted into a 20-ml dilution vial and nine mls of the appropriate solvent is added from a graduated pipette. The vial is capped and the sample is mixed thoroughly.

The sample is now ready for fusion and measuring the fluorescence in accordance with the procedures described on Pages 49 - 51.

## Extraction

One 20-ml extraction vial for each extracted blank, each standard, and each sample is removed from the storage drawer. For inventory or special samples, two vials are used for each. These vials are marked by a crayon pencil or permanent ink pen with the sample requisition number, the concentration of the standard, or the blank.

A 1-ml, 5-ml, or 10-ml portion of the sample is transferred with a Normax pipette into the appropriate vial.

Since the uranium concentration of the samples received for fluorimetric analysis ranges from depleted to highly enriched levels, it is not feasible to include the detailed extraction information for each individual type sample in this procedure. Tables which indicate the sample material type, the milliliters of sample, standard, or water for blank, the milliliters of concentrated $HNO_3$ or 1 M $Al(NO_3)_3$ in 1 M $HNO_3$, and the milliliters of TOPO-varsol solution (extractant) to be used as well as the calculated dilution factors are readily available in the fluorimetric area of the laboratory. In general, the following applies for the aqueous liquid samples:

Product-level condensates and raffinates usually have low uranium concentrations and require greater precision and accuracy than other sample types in the low range of fluorimetric determination. Consequently, the uranium content of these samples is concentrated by extracting 10 mls of sample with two mls of TOPO-varsol solution.

Other sample types usually have higher uranium concentrations and can be extracted by a 1:1 ratio of sample to solvent (five mls of sample and five mls of TOPO-varsol solution). Some samples, such as feeds and aqueous products, have high uranium concentrations and the uranium content is diluted by extracting 1 ml of sample with 10 mls of TOPO-varsol solution.

After the addition of the sample to the vial, the appropriate amount (usually five mls) of concentrated $HNO_3$ or 1 M $Al(NO_3)_3$ in 1 M $HNO_3$ is added with a graduated pipette and the appropriate amount (usually five mls) of TOPO-varsol solution is added with a transfer-type pipette. The addition of nitric acid adjusts both the acid and nitrate concentration for better extraction. For some samples, aluminum nitrate monohydrate is used as a source of nitrate and hydrogen ions. The aluminum possesses the added advantage of serving to complex ions that interfere with extraction such as fluoride, sulfate, and phosphate.

The vials are capped, placed in a horizontal position in the mechanical shaker, and shaken for 10 minutes.

After shaking 10 minutes, the vials are removed and allowed to stand for one minute for the layers to separate. The samples are now ready for fusion as described in the section that follows.

## Fusion

Usually a group of ten samples are processed together. At the beginning of each shift operation, two blanks, two standards, and six samples are processed together. Inventory

and certain special samples require two independent determinations, thus only half as many of these are processed together.

A Lavite tray is selected, see description on Page 40. Clean platinum dishes are removed with forceps from the storage container and one dish is placed in each well of the first two rows of the tray. Each row of two platinum dishes represents either one blank, one standard, or one sample, except in cases of inventory samples or other special samples when two rows will be used.

The Lavite tray containing the platinum dishes is placed under infrared lamps to dry the dishes.

When the dishes are dry, the Lavite tray is removed from under the infrared lamps and, with a pelletizer, a 0.2-gram portion of flux (2% lithium fluoride in sodium fluoride) is placed in each dish.

With the short end of a 100-lambda pipette inserted into a pipette syringe, the pipette is immersed in the top or organic layer of the extracted sample in the vial. The pipette must not enter the lower or aqueous layer. If the sample was prepared by dilution, the sample solution in the vial or volumetric flask must be mixed thoroughly before the pipette is immersed. Using the syringe, the sample is pulled up into the pipette until it reaches the calibration mark. As the pipette is removed from the vial or flask, the tip is wiped across the lip of the vial or flask. The tip of the pipette is held close to the top of the first pellet in the row representing the particular sample; the plunger is pressed until all the sample is forced out onto the pellet. The operation is repeated for each dish using the same pipette for each dish on the same sample and a clean pipette for each sample.

The Lavite tray containing the platinum dishes and samples is placed under infrared lamps and the samples are allowed to dry for 25 to 30 minutes or until "smoking" ceases. The lamps should be approximately six inches above the dishes. When the samples are dry, the tray is removed from under the lamps.

Using forceps, the platinum dishes are removed systematically from the Lavite tray and placed on a Nichrome wire screen. The exact order of the dishes on the screen must be remembered since they must be returned to the Lavite tray in the same order.

The propane to the modified Fletcher burner is turned on and ignited. The pressure of the propane is then adjusted to an approximate setting of 45 on the propane rotameter and the compressed air pressure is adjusted to a setting of 9.5 on the air rotameter. Using tongs, the Nichrome wire screen with the platinum dishes is lifted onto the top of the burner. The pellets will start to melt within one-half minute and have a clear melt within one or two minutes. It is important that the melt be completely clear. If there is cloudiness in the

melts after two minutes, the pressure of the compressed air is increased. Any excessive noise in the burner is eliminated by reducing the pressure of the air. If a clear melt has not developed after two minutes, the samples are allowed to stand until a clear melt does develop. The clear melt is held for one minute then the air pressure is reduced until the flame begins burning above the Nichrome screen. This is indicated by black splotches appearing on the red hot screen. The air pressure is increased until the splotches just disappear. The samples are allowed to remain on cooling flame for one minute.

Using tongs, the Nichrome screen and samples are removed from the burner, placed on a metal plate, and allowed to cool for 10 minutes. The platinum dishes are then placed on the Lavite tray in their original order. The samples are allowed to cool an additional 10 minutes before the fluorescence is measured.

Measuring the Fluorescence

The fluorescence intensity of each dish is measured.

## CALCULATIONS

Micrograms of Uranium per Milliliter of Sample

Extraction or Dilution Method - Formula for μg U/ml on volume basis:

$$\mu g\ U/ml = (\text{avg reading} - \text{blank})\ (\text{machine factor})\ (\text{dilution factor}^{(a)})$$

If the μg U/ml is below 1.0, the result is rounded to two decimal places. If the μg U/ml is 1.0 or above, the result is rounded to one decimal place.

Grams of Uranium per Gram of Sample

Formula for g U/g if specific gravity measurement is used:

$$g\ U/g = \frac{\mu g\ U/ml \times 10^{-6}}{\text{specific gravity}}$$

---

(a) Dilution factor for the extraction method $= \frac{\text{ml of extractant}}{\text{ml of sample}}$

Dilution factor for the dilution method $= \frac{\text{ml of sample + ml of diluent}}{\text{ml of sample}}$

Formula for g U/g if a weight portion of the sample is taken and diluted to volume:

$$g\ U/g = \frac{\mu g\ U/ml \times volume \times 10^{-6}}{sample\ weight}$$

Formulas for g U/g Meltemp oil sample:

$$g\ U/g\ (aqueous) = \frac{\mu g\ U/ml \times volume \times 10^{-6}}{sample\ weight}$$

$$g\ U/g\ (organic) = \frac{\mu g\ U/ml \times volume \times 10^{-6}}{sample\ weight}$$

Total g U/g Meltemp oil = g U/g (aqueous) + g U/g (organic)

Grams of Uranium per Liter of Sample

Formula for g U/l:

$$g\ U/l = \frac{\mu g\ U/ml \times volume \times 10^{-6}}{sample\ weight} \times 1000$$

Micrograms of Uranium per Liter of Sample

Formula for µg U/l:

$$\mu g\ U/l = g\ U/l \times 1000$$

SAMPLE WORK SHEET AND CARD

Description and Explanation of Fluorimetric Analysis Data Sheet

Fluorimetric analysis data sheets (Figure 10) are provided for recording and calculating the fluorimetric data. A carbon copy is attached to the original so that all data are recorded at the same time; the two sheets are separated and independent calculations made.

The spaces at the top of the sheet are for the following information:

Average Blank Reading - Values for the "direct blank" and the "extracted blank" are recorded in this space.

Sheet Number 1753

## FLUORIMETRIC ANALYSIS

Average Blank Reading: Direct 1.0; Extracted 0.7

Machine Factor: .0098

Standard Concentration: .05 µg U/ml

Date: 7/22/58

Tray Number: 7

Started By: SHC Read By: SHC

Cal. By: SHC Ckd. By: CB

Appvd. By: JCT Rptd. By: MKH

µgU/ml or ppm. = Av. Reading − Av. Blank x Machine Factor x Dilution Factor

| Anal. No. | Req. No. | Sample Prep. Mls. Samp. | Mls. Ext. | Mls. Diluent | Fluorophotometer Readings, µa | Av. Read. | Net Read. | Net Read. x Macn. Fact. | Dilution Fact. | Ans. µgU/ml or ppm |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Direct Blank | | | | 1 0.9 2 1.2 | 1.0 | | | | |
| 2 | Extracted Blank | 5 ml H2O | 5 | | 1 0.8 2 0.6 | 0.7 | | | | |
| 3 | .05 Std. | 10 | 2 | | 1 31.0 2 23.0 | 27.0 | 26.3 | 0.26 | 0.2 | 0.05 |
| 4 | 111701 | 5 | 5 | Ext. | 1 465.0 2 480.0 | 472.5 | 471.8 | 4.62 | 1 | 4.6 |
| 5 | 111702 | 10 | 2 | | 1 38.0 2 35.0 | 36.5 | 35.3 | 0.35 | 0.2 | 0.07 |
| 6 | 798151 | 1 | | 9 | 1 105.0 2 125.0 | 115.0 | 114.0 | 1.12 | 10 | 11.2 |
| 7 | 802308 | Direct | | | 1 280.0 2 305.0 | 292.5 | 291.5 | 2.86 | 1 | 2.9 |
| 8 | | | | | 1 2 | | | | | |
| 9 | | | | | 1 2 | | | | | |
| 10 | | | | | 1 2 | | | | | |

REMARKS: Calculation of Gram Uranium/Gram Using Specific Gravity Measurement: (798151)

$$\frac{\mu g\ U/ml \times 10^{-6}}{sp.gr.} = \frac{11.2 \times 10^{-6}}{1.085} = 0.000010 \text{ g U/g sample}$$

Calculation of Gram Uranium/Gram Using Weight Portion of Sample Diluted to Volume: (802308)

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$$\frac{\mu g\ U/ml \times volume \times 10^{-6}}{wt.\ of\ sample\ taken} = \frac{2.9 \times 1000 \times 10^{-6}}{99.7} = 0.000029 \text{ g U/g sample}$$

Figure 10. TYPICAL FLUORIMETRIC ANALYSIS DATA SHEET.

Machine Factor – The machine factor or calibration factor is recorded in this space.

Standard Concentration – The uranium concentration in the standards processed during the particular shift is recorded in this space.

Date – The date on which the samples are analyzed is recorded in this space.

Tray Number - The number of the Lavite tray on which the samples were processed is recorded.

Started By, Read By, Calculated By, Checked By, Approved By, and Reported By - The initials of all analysts either processing the particular samples, recording the data, or calculating the results as well as the laboratory supervisor approving and reporting the results are recorded in the appropriate spaces.

The center section of the data sheet is divided into ten rows and nine columns with the third column subdivided into three additional columns.

Analysis Number - This column numbers the rows in consecutive order and is the order in which the samples are processed and the data recorded.

Requisition Number - The requisition number for each sample or blank is recorded in this column.

Sample Preparation; Subdivision: Mls Sample, Mls Extractant, and Mls Diluent - The volume of the sample and the volume of the organic extractant or the volume of the diluent used are recorded in the appropriate subdivisions of this column. Only the volume of the solutions transferred to the 20-ml extraction or dilution vials is recorded here. Any previous dilution or concentration of the sample is recorded on the master requisition or other work cards provided.

Fluorophotometer Reading, μa - The fluorophotometer reading in microamperes on each of two dishes prepared for each sample is recorded in this column.

Average Reading - The average of the readings of the two dishes on each sample is calculated to one decimal place and recorded in this column.

Net Reading - The net reading, which is the difference between the average of the fluorophotometer reading for the sample and the average blank reading, is calculated to one decimal place and recorded in this column. If the sample is run direct or diluted, the direct blank is used. If a sample is extracted, the extracted blank is used.

Net Reading x Machine Factor - The product of the net reading multiplied by the machine or calibration factor is calculated to two decimal places and recorded in this column.

Dilution Factor - The appropriate dilution factor calculated by the method described on Page 51 is recorded in this column.

Microgram Uranium/Milliliter - The appropriate μg U/ml as calculated by the method described on Page 51 is recorded in this space.

The space at the bottom of the sheet is provided for making additional calculations such as: gram uranium/gram, gram uranium/liter, and microgram uranium/liter (see Pages 51 and 52).

After all calculations have been made, the master requisition card for each sample is stamped with one of two forms. One form (Figure 11) is for all samples that require a microgram-of-uranium-per-milliliter answer and the other form (Figure 12) is for samples requiring a gram-of-uranium-per-gram-of-sample answer or any other. The master requisition cards and the original data sheets are transferred to the records office.

Answer _____ μg/ml

Sheet No. _____

Date _____

By _____

Figure 11. MASTER REQUISITION CARD. (For Samples That Require a Micro-gram-of-Uranium-per-Milliliter Answer)

Answer _____ g/g

Sheet No. _____

Date _____

By _____

Figure 12. MASTER REQUISITION CARD. (For Samples That Require a Micro-gram-of-Uranium-per-Gram-of-Sample Answer)

## Explanation of Miscellaneous Laboratory Work Card

A miscellaneous laboratory work card (Figure 13) is used for all fluorimetric samples that require sample preparation or two independent determinations to make a complete analysis. This places all the data on one card and thus eliminates checking through several fluorimetric analysis data sheets in order to check the data and calculations. A Meltemp oil sample is an example of this type of analysis.

The requisition number, recycle, date, and container number are recorded in the appropriate spaces at the top of the card. All calculations for the aqueous portion and the organic portion are indicated on the card. The sum of the g U/g in the aqueous phase and the g U/g in the organic phase equals the total g U/g Meltemp oil. The number of the fluorimetric analysis data sheet where each portion was analyzed is recorded on the card.

## MISCELLANEOUS LABORATORY WORK CARD

| 1999 | 11/17/58 | 75 | 1706-47 | "Meltemp" Oil g U/g | 888888 |
|---|---|---|---|---|---|
| RECYCLE | DATE | CONT NO | BATCH NUMBER | DETERMINATION | REQUISITION NUMBER |

Sample Weight 129.0 g

| | | |
|---|---|---|
| Sample Portion | Organic | Aqueous |
| Sheet Number | 106 | 108 |
| Method | Direct | Extracted 5:5 |
| Volume | 2000 ml | 100 ml |

CALCULATIONS

$$\frac{9.9 \times 2000 \times 10^{-6}}{129.0} = 0.000153 \text{ g U/g (organic)}$$

$$\frac{22.0 \times 100 \times 10^{-6}}{129.0} = 0.000017 \text{ g U/g (aqueous)}$$

REPORT:

Total g U/g "Meltemp" Oil = 0.000170

LAB-99 (7-58)

Figure 13. WORK CARD WITH TYPICAL GRAMS-OF-URANIUM-PER-GRAM-OF-SAMPLE DATA FOR MELTEMP OIL.